INJECTION GROUTS FOR MOLASSE SANDSTONES: PRELIMINARY ASSESSMENTS

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Abstract

Selected proprietary injection grouts as well as "home-made" formulations have undergone laboratory and limited field testing in order to find a solution for back-filling large voids in molasses sandstone affected by severe contour scaling. Although good working properties have generally been found, performance characteristics often fall short of specific performance criteria. Lime-based grouts, despite present important shortcomings, are all the same considered most promising for further development and field-testing.

1. Introduction

Lausanne cathedral, built during the 13th century, is one of Switzerland's most important medieval monuments. Surprisingly, its exterior façades, composed largely of Aquitanian molasse sandstone, still conserve up to 30% of original building stone. One of the most severe weathering phenomena encountered on these surfaces is the formation of thick contour scales, giving rise to friable sub-surface zones which develop into sometimes large voids situated at depths ranging from a few millimetres to up to 10cm, with consequent risk of detachment of significant areas of historic stone surfaces. Although considerable replacement of stone has taken place on the flying buttresses for structural reasons, it is intended to conserve most of the weathered stones on the façade elevations and to devise for the deep contour scaling a suitable treatment procedure. With this aim in view, we have been carrying out trials with different injection grouts. Although commercially available products based on ethyl silicate may have seemed appropriate at first and have been included in the present study, other formulations, both commercial and "home-made", were considered; preliminary testing of the materials took place in the laboratory in order to determine some of their characteristics and to narrow down the choice for subsequent field-testing. Both working properties and performance characteristics were assessed and compared with what was considered to be desirable. An important precept to the investigation was that the injection material should have similar mechanical properties to the weathered molasse sandstone in order to prevent failure between the two materials under natural weathering; consequently, petrophysical tests were carried out on samples of weathered stone as well as on the different grouting materials.

2. Choice of materials

Table 1: type and composition of test formulations

Designation	Binders	Fillers
FUNCOSIL	29ml FUNCOSIL KSE 500 STE 71ml FUNCOSIL KSE 300 E	64g FUNCOSIL KSE Filler A 40g FUNCOSIL KSE Filler B 26g glass spheres (<50µm) 5g green earth pigment
SYTON	SYTON X30	2 vol. quartz powder (<0-63µm) 2 vol. molasse powder (<0-400µm) 1 vol. fumed silica (ACEMAT HK 125)
Lime + GB	1 vol. dispersed lime 2 vol. water	1 vol. Scotchlite glass bubbles (<0-177µm) 3 vols. molasse powder (<0-400µm)
Lime + GB + pozz.	1 vol. dispersed lime 2 vol. water	1 vol. Scotchlite glass bubbles (<0-177µm) 1.5 vol. local sand (<0-1000µm) 1.5 vol. terra pozzuolana (<0-1000µm)
LEDAN	2 vol. LEDAN TC1 PLUS 1.5 vol. water	
PLM-M	3 vol. PLM-M 2 vol. water	1 vol. molasse powder (<0-400µm)

Six grout formulations were tested (table 1). The materials fall into 3 categories: **ethyl silicate-based grouts**, comprising ethyl silicate binders with reportedly high gel deposits, mixed with inert fillers (designated FUNCOSIL and SYTON); **lime-based grouts**, comprising a dispersed lime binder mixed with either inert fillers or inert and pozzolanic (pozz.) fillers; and commercial **hydraulic grouts** with unspecified components (LEDAN and PLM-M). The materials were chosen on the basis of reported or published experience on grouts used in the field of stone conservation [1], or on manufacturers literature; the use of dispersed lime in grout formulations has already been reported [2], but we also benefited from experience transmitted to us through the School of conservation in Berne. The use of Scotchlite glass bubbles (GB) in grout formulations has also been reported elsewhere [3].

3. Preparation of samples

Porous sample supports made of sandstone were preferred to non-porous supports so as to reproduce as far as possible real-life setting conditions; not only can water and ions released during setting travel through the porous system as they would in a real wall, but the suction provided by the porous support permits faster drying, an important *in situ* factor which has a major influence on the strength of the hardened grout [4, 5].

Initially, to facilitate the evaluation and comparison of results, identically sized molasse sandstone blocks were hollowed out to form a reservoir 4cm deep by 4cm wide by 8cm long, into which the grout was injected via a syringe. The size of the reservoir was imposed by the size of the grout sample required for the laboratory testing procedures; although these sample containers were useful in testing certain working and performance characteristics, it was clear that the grouts would not, in real life, be expected to fill such spaces.

In order to better reproduce grouting conditions, a second series of supports was prepared using “window boxes”, designed to visualise flow capacity and other working properties of the grouts. The window boxes were made by sandwiching between a sandstone block and a perspex window a tortuous path along which the injected material was intended to flow; the space between the sandstone and the perspex window was about 2cm, simulating the type of void we would expect to find in real-life; occasionally, loose material was placed along the path to test penetration qualities. During application and setting of the grouts, working properties could be visually recorded; after setting, the perspex windows were peeled off and 1.5cm large, 1.5cm thick and 4cm long samples were cut out for the laboratory tests.

Further tests were carried out on cubes of molasse (4cm edge) cut from badly deteriorated 19th century replacement stone, which had undergone consolidation with the ethyl silicate WACKER OH 100 (contour scaling is always accompanied by areas of friable stone which need first to be reinforced if grouting is to be successful). The majority of these cubes were used to test the petrophysical properties of the weathered and consolidated (W&C) molasse, but a few of them were also used to test adhesion between the W&C molasse and the grouts by simple end-bonding using the grout mixtures.

Finally, limited field tests were carried out on ashlars presenting real-life contour scaling. After setting of the injected material, the test zones were sawn to observe, in cross-section, the degree of flow and penetration of the grout. Samples of dried grout taken from these areas were too small (0.5 to 1cm thick) to be used for tests on performance characteristics, but they were very useful in observing working properties.

4. Assessment methods: rationale

4.1 Working properties

Given that an injection grout should, by nature, allow introduction into a cavity by means of a syringe, a **low viscosity** is obviously an important property to enable good

flow capacity. On the other hand, low viscosity can increase the risk of shrinkage and crack formation during setting.
The **injectability** of a grout is closely associated with viscosity, but can also be influenced by grain size of fillers and the speed of "coagulation" of the injected material.
Setting time is an important factor given that the grout should remain fluid long enough to fill the farthest reaches of the cavity before solidification; this property was found to be particularly important during the field tests, where repeated injections over several hours were necessary in order to completely fill the voids.
These working properties, often closely interrelated, were assessed on the basis of empirical observations during preparation of the mixtures, as well as before, during and after their introduction into the different sample supports; the window-boxes were particularly useful in this respect.

4.2 Performance characteristics

Formation of **shrinkage** cracks during drying and hardening of the injected material is clearly a negative factor since such behaviour can compromise good adhesion of the mortar to the cavity walls and limit water transfer through the alteration profile.
To provide sufficient support for the detached areas of stone good **adhesion** between the weathered stone and the injection grout is considered an essential factor. Adhesion was tested empirically by recording the ease with which the samples could be detached from the sample supports, as well as on the grounds of end-bonding capacities.
The grout layer introduced between the detached stone surface and the main stone bloc should not act as a water reservoir or as a water barrier. In both cases, durability and performance of the system would be negatively affected. Porosity compatibility between the grouts and the W&C molasse sandstone was tested by means of laboratory measurements of **total porosity** and **capillary absorption kinetics** (according to RILEM recommendations [6].
The injection grout must be strong enough to support the detached areas of surface stone, some of which can be relatively heavy (several kilos), although additional support is achieved by means of edging repairs. Furthermore, in exposed conditions, the grout will have to resist dimensional changes of the sandstone following temperature and humidity variations: tensile strength should therefore be good. As tensile strength can be deduced from **compressive strength** measurements (typically 20 to 50 times more than tensile strength), only compressive strength was measured on the grouts and the W&C molasse according to the RILEM recommendations [6].
Porous materials can swell when the relative humidity and/or the water saturation increases. A too high dilatation would be unacceptable because it could provoke the detachment of the re-adhered section of the stone. As hygric dilatation is less than **hydric dilatation**, we measured only the latter on the mortars and on the W&C molasse (according to the RILEM recommendations [6]).
The capacity of porous materials to **adsorb water vapour** is a critical factor on exposed surfaces. If adsorption is too high, during periods of high relative humidity, liquid water can form in the porous network of the material due to capillary condensation; if the

grouting or adjoining materials contain soluble salts, subsequent damage can rapidly appear due to more frequent salt dissolution and crystallization cycles than in materials with low water vapour adsorption. Equilibrium water vapour adsorption of the mortars was measured by placing samples in 93% relative humidity at room temperature. Unfortunately, measurements for the W&C molasse are not yet available and could thus not be compared during the present study.

4.3 Salt content

The dissolution and crystallization of soluble salts during wetting and drying cycles being one of the most important causes of stone deterioration, it is clearly desirable that injection grouts should be free of these salts. Salt analysis was carried out after aqueous extraction of the soluble phases from some of the selected grouts and from the W&C molasse sandstone. The ion content was then measured on the solution by means of ion chromatography according to the procedures described by Blaüer Böhm [7]. Salt content was only measured on the hydraulic PLM-M and LEDAN grouts, since previous testing on similar hydraulic materials had demonstrated high salt content [8, 9, 10]. As it was clear that the weathered molasse probably had high soluble salt content, analysis of this material was also carried out for comparison.

4.4 Maturation

Due to time constraints, all the test grouts were analysed before full maturation. However, in order to determine the effect of maturation on performance characteristics, a batch of the two hydraulic grouts PLM-M and LEDAN underwent artificial maturation to compare results with the fresh samples. Samples of the grout were hydrated during 72 hours at 80°C in a very high relative humidity; they were then placed in a carbon dioxide enriched atmosphere at room temperature and high relative humidity to accelerate the carbonation process. This carbonation was continued until the phenolphthalein test proved that all the samples were perfectly carbonated.

5. Results

5.1 Working properties

Table 2: working properties. +++ = very good; ++ = good; + = fairly good; - = bad

	Viscosity	Injectability	Setting time
FUNCOSIL	+++	+++	+++
SYTON	++	+++	++
Lime + GB	+	++	-
Lime + GB + pozz.	+	++	-
LEDAN	++	+++	+
P LM-M	++	+++	+

The working properties of the tested mixtures are summarized in table 2.

- **Viscosity** of the six grouts was generally good, with good flow capability within the window boxes. The LEDAN, PLM-M, FUNCOSIL and SYTON grouts all flow easily along the tortuous path of the window boxes and fill all the voids. However, only the FUNCOSIL grout was able to penetrate well into the small piles of loose sand distributed within the boxes, although limited penetration was achieved by the hydraulic grouts; the SYTON grout did not penetrate at all into the loose sand. The lime-based grouts were generally more viscous and some pressure had to be exerted to fill all the voids; no penetration was achieved into the loose sand particles. Similar observations were made during the field tests, with no significant differences in the filling capacity of the FUNCOSIL, SYTON and lime + GB formulations.

- All the grouts showed good **injectability**. For the lime-based grouts, due to filler size, syringe needles could not be smaller than 2mm in diameter.

- The FUNCOSIL grout has the longest **setting time**, permitting repeated wet in wet injections and topping up. The SYTON mixture has similar working properties. The hydraulic grouts, on the other hand, set more quickly on the surface, although remain liquid during several hours in deeper zones. The lime-based grouts tend to harden in the entry channels, making repeated injections more difficult.

5.2 Qualitative performance characteristics

Table 3: qualitative performance characteristics. + = no shrinkage / good adhesion; - = no adhesion / a few cracks; -- and --- = more and more cracks.

	Shrinkage	Adhesion
FUNCOSIL	-	+
SYTON	--	+
Lime + GB (non-carbonated)	+	+
Lime + GB + pozz. (non-carbonated)	---	-
LEDAN (fresh)	+	+
PLM-M (fresh)	+	+

Shrinkage and adhesion characteristics were measured visually (table 3). Cracks formed readily on the ethyl silicate grouts during setting, although good adhesion was achieved; the FUNCOSIL grout generally performed better than the SYTON grout, which showed sometimes severe disruption due to shrinkage. Few shrinkage cracks were observed on the lime + GB grout and good adhesion was also recorded. Subsequent tests on lime-based formulations mixed with pozzolanic fillers showed poor shrinkage and adhesion properties, with considerable disruption of the samples. The hydraulic grouts, on the other hand, showed good shrinkage and adhesion properties, making it sometimes impossible to separate the grout samples from their containers without sawing. Most of the grouts were able to successfully adhere two pieces of stone together and presented sufficient resistance when pulled; only the lime + GB + pozz.

behaved poorly in this respect, probably due to the severe cracking during setting The poor performance of the lime + GB + pozz. mixture made it useless to continue testing and this material was abandoned until preparation procedures had been reviewed and more stable formulations devised.

5.3 Quantitative performance characteristics

Table 4: quantitative performance characteristics. Tp = total porosity (%) – Cp = capillary porosity (%) – S = Hirschwald coefficient = Cp/Tp x 100 (%) – Coef A = mass coefficient of capillarity ($mg/cm^2.h^{1/2}$) – Coef B = linear coefficient of capillarity – ε72 = hydric dilatation coefficient (mm/m) – Vads = water vapour adsorption at a 93% relative humidity = mass of adsorbed water vapour at 93%RH / mass of the dry sample x 100 (%) – CS = compressive strength of dry samples (N/mm^2)

	Tp	Cp	S	Coef A	Coef B	ε72	VAds	CS
FUNCOSIL	42	36	85	177	0.6	1.4	2.1	4
SYTON	35	29	84	353	1.0	1.2	4.8	5
Lime + GB non-carbonated	40	27	68	347	1.4	0.8	1.4	1
LEDAN non-matured	54	44	82	38	0.1	3.1	24	16
LEDAN matured	46	40	86	28	0.1	2.3	8	20
PLM-M non-matured	46	42	92	59	0.2	2.6	12	11
PLM-M matured	40	36	90	93	0.3	2.6	6	20
W&C molasse sandstone	19	11	64	94	0.7	2.5	-	12

- The **total porosity** (Tp) is high for all the grout mixtures. It varies from 35% (SYTON) to 54% (LEDAN non-matured). These values are all much higher than the porosity of the W&C molasse samples (around 19%).

- The linear (B) **coefficients of capillarity** of all the grouting mixtures is generally of the same order as the sandstone, excepting the hydraulic binders (LEDAN and PLM-M), where B is considerably lower compared to the sandstone sample values. But as we can see with the mass (A) coefficient, the capillary kinetics of PLM-M (59 or 93 $mg/cm^2.h^{1/2}$) and LEDAN (38 and 28 $mg/cm^2.h^{1/2}$), matured or not, are slower than for the W&C sandstone samples (94 $mg/cm^2.h^{1/2}$), whereas SYTON (353 $mg/cm^2.h^{1/2}$), FUNCOSIL (177 $mg/cm^2.h^{1/2}$) and lime + GB (347 $mg/cm^2.h^{1/2}$) are much faster. If we consider the **maximum capillary saturation** (Cp), we note that it is high for all of the mixtures; the Hirschwald coefficient (S), which represents the percentage of the pore volume filled with water at the end of the capillary absorption, is higher than 50% for

all the grouts. The smallest percentage value was measured on the lime + GB (68%) and the highest was obtained for the fresh PLM-M (92%).
- The best **hydric dilatation** coefficients at 72 hours (ε72) were obtained for the lime-based grout (1 mm/m), SYTON (1.2 mm/m) and FUNCOSIL (1.4 mm/m). On the other hand, hydraulic grouts have high ε72 : between 2.3 and 3.1 mm/m.

- The weight percentage of **water vapour adsorption** of the non-matured hydraulic grouts is far too high: 12% for non-matured PLM-M and 24% for non-matured LEDAN. Even if these values were reduced by maturation, they remained relatively high (6% for matured PLM-M and 8% for matured LEDAN). The ethyl silicate-based grouts have average values of water adsorption (4.8% for SYTON and 2.1% for FUNCOSIL), while the lime + GB has the lowest water adsorption (1.4%).

- LEDAN and PLM-M obtained the highest values of **compressive strength**, respectively 16 and 11 N/mm^2 (non-matured) and 20 N/mm^2 for both when matured. The lime mortars performed worst, obtaining only 1 N/mm^2, while the ethyl silicates obtained values between 4 and 5 N/mm^2.

5.4 Salt content

Table 5: salt content. In µg/g. nd = not detected, ss = saturated solution because of the selected extracting procedure [6]

	Na+	NH4+	K+	Mg++	Ca++	F-	Cl-	NO3-	PO4--	SO4--
LEDAN non- matured	648	nd	2072	nd	ss	114	29	14	11	2421
LEDAN matured	234	nd	342	212	5403	117	49	nd	nd	5879
PLM-M non- matured	1603	nd	364	nd	ss	65	25	22	72	1549
PLM-M matured	1677	nd	87	153	2623	67	37	35	14	30
W&C molasse	71	12	396	78	3031	nd	nd	24	nd	8220

The salt content was not measured for the FUNCOSIL, SYTON and lime grouts, but only for the proprietary hydraulic grouts LEDAN and PLM-M. Ion concentrations remained high even after maturation, as we can see in table 5. The Ca++ content decreased considerably during the carbonation: in the fresh samples Ca contents were on the level of saturated Ca(OH)2-solutions, which decreased to 5403 µg/g (LEDAN) and 2623 µg/g (PLM-M). The high SO4-- content of both mixtures increased during the maturation but Na+ has a high concentration in the PLM-M whatever the maturation state (1603 and 1677 µg/g respectively before and after maturation). Mg++ is released

in small quantities during the maturation as the K+ content decreased for both LEDAN and PLM-M.
As we can see in table 5, some of the ion concentrations of the grouts are still lower than those of the W&C molasse, which contains a high gypsum concentration (8220 µg SO4--/g, 3031µg Ca++/g) in addition to some potassium, magnesium and sodium sulphates (396 µg K+/g, 78 µg Mg++/g and 71 µg Na+/g).

6. Discussion

All the grouts have good working properties; they have low viscosity permitting easy injectability via syringe and good flow capacity, although the lime grout's ability to flow into the farthest reaches of the voids was more limited than the ethyl silicate and hydraulic grouts. For all the grouts, setting times seemed long enough to complete the filling process, although once again the lime grouts would probably set too fast for complex voids requiring long filling periods.

Performance characteristics are generally less satisfying: excepting the lime + GB + pozz. grout, all the mixtures show good adhesif power; however, excepting the commercial hydraulic and lime + GB grouts, all the other displayed high shrinkage, with extensive crack formation. With the ethyl silicate grouts, this was to be expected since it is generally recognised (by the manufacturers as well as experienced conservators) that these materials are not designed for filling voids larger than 5mm thick. Although lime-based grouting mixtures have been commonly used to fill large voids behind wall paintings, we have so far not been able to obtain grouts which do not crack on setting; the initial positive results with light and bulky fillers such as glass bubbles could not be reproduced in subsequent tests.
As yet too little is known about water transfer at the stone/grout interface: fast capillary kinetics could be a major disadvantage if the injection grouts become water saturated and drying is impeded. On the other hand, slow capillary kinetics could also be problematic since too low water absorption by the grout could lead to too high water content in the detached surface stone. To be sure that the water transfer at the stone/grout interface is good, field experiments should be done.
According to the Hirschwald theory [11], a porous material with a Hirschwald coefficient higher than 85% is frost susceptible. In this case, we can suppose that all the tested recipes are frost susceptible, except for lime + GB and perhaps SYTON and FUNCOSIL.
If it is admitted that a hydric dilatation coefficient at 72 hours higher than 1 mm/m is dangerous, then lime-based, SYTON and FUNCOSIL grouts perform best.
Only the hydraulic grouts (LEDAN and PLM-M) displayed high enough compressive strength values, although more testing needs to be done on lime mixtures containing pozzolanic fillers intended to improve strength. On the other hand, too high compressive strength may enter into conflict with another criterium, namely that the grout should offer similar or lower strength compared with the original material with

which it is in contact, otherwise differential movements between the two in out-door conditions could accelerate detachment.

The high salt content of the hydraulic grouts, even if this is sometimes lower than the salt content of the weathered molasse, falls far short of the accepted principle that conservation materials should not introduce soluble ions into porous materials as they could form hygroscopic salts [12] that can destroy both the grout itself and the W&C molasses sandstone. Despite this, given that the hydraulic grouts performed better than the other grouts in terms of key criteria such as compressive strength, adhesion and shrinkage, it was decided to pursue field tests with this material. It is clear however that future development will weigh in favour of the lime-based home-made grouts with reduced risk of salt contamination.

7. Conclusion

This study give important information on the properties of the conservation materials tested and their adaptability on exterior exposed historic masonry; further information will be gathered in the coming months after inspection of the limited field tests. Because of the high salt content of the hydraulic grouts and the incompatibility of the ethyl silicate grouts with large voids, only the lime-based grouts remain open, in our minds, for further development. Reported experience elsewhere with dispersed lime binders for back-filling stone has so far given promising results [2], which comforts our choice of this material; inert fillers such as glass bubbles gave promising initial results in compensating the high shrinkage associated with lime-based materials and it would seem desirable to incorporate this filler into future recipes. It would also seem desirable to incorporate pozzolanic materials into future test grouts in order to improve compressive strength, although salt content of these materials should be analysed. Experimentation could also be carried out on other means of compensating shrinkage, such as those reported by Smith [3] (for example, the use of aluminium powder); other means of improving fluidity and lengthening setting time could also be investigated.
In parallel to these further investigations, we feel it is worth questioning the validity of the performance criteria and the laboratory testing procedures designed to measure them. It is often considered that a conservation material can never meet ideal criteria. What, then, can be considered acceptable criteria? For example, if the stone already contains large amounts of soluble salts, how dangerous is it to introduce a material with high salt content which otherwise presents excellent qualities?

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